



SLOVENSKI STANDARD

SIST EN 13211:2002

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Kakovost zraka - Emisije nepremičnih virov - Ročna metoda za določevanje koncentracije celotnega živega srebra

Air quality - Stationary source emissions - Manual method of determination of the concentration of total mercury

Luftqualität - Emissionen aus stationären Quellen - Manuelles Verfahren zur Bestimmung der Gesamtquecksilber-Konzentration

Qualité de l'air - Emissions de sources fixes - Méthode manuelle de détermination de la concentration en mercure total

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

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English version

Air quality - Stationary source emissions - Manual method of determination of the concentration of total mercury

Qualité de l'air - Emissions de sources fixes - Méthode manuelle de détermination de la concentration en mercure total

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This European Standard was approved by CEN on 6 January 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 264 "Air quality", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2001, and conflicting national standards shall be withdrawn at the latest by July 2001.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European standard specifies a manual reference method for the determination of the mass concentration of mercury in exhaust gases from ducts or chimneys. This European standard is validated for the determination of the mass concentration of total mercury in exhaust gases from the incineration of waste for the concentration range of total mercury from 0,001 mg/m³ to 0,5 mg/m³¹⁾. The method may be applicable for exhaust gases from other sources with the following typical composition:

total suspended matter	from 0 mg/m ³ to 20 mg/m ³
C _x H _y	from 0 mg/m ³ to 10 mg/m ³
HCl	from 0 mg/m ³ to 50 mg/m ³
HF	from 0 mg/m ³ to 10 mg/m ³
SO ₂	from 0 mg/m ³ to 250 mg/m ³
CO	from 0 mg/m ³ to 250 mg/m ³
NO _x	from 0 mg/m ³ to 500 mg/m ³
CO ₂	from 0 % (volume fraction) to 15 % (volume fraction)
H ₂ O (g)	from 2 % (volume fraction) to 25 % (volume fraction) (actual)
O ₂	from 8 % (volume fraction) to 15 % (volume fraction) (dry, actual)
temperature	from 60 °C to 140 °C

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1483	Water quality — Determination of mercury. https://standards.iteh.ai/catalog/standards/sist/04b71585-8bb4-4099-9672-cc7a243aca/sist-en-13211-2002
prEN 13284-1:1998	Stationary source emissions — Determination of low range mass concentrations of dust — Part 1: Manual gravimetric method.

3 Terms and definitions

For the purpose of this European Standard, the following terms and definitions apply:

3.1

mercury

mercury and mercury in its compounds

3.2

total mercury

sum of the mercury in exhaust gas independent from the state (gaseous, solved in droplets, solid, absorbed on particles)

3.3

representative sampling

isokinetic, flow equivalent sampling at the required minimum number of sampling points in the sampling plane as stated in the prEN 13284-1:1998

¹⁾ m³ expressed as m³ under dry conditions, normalized to 0 °C and 101,325 kPa and at 11 % (volume fraction) O₂ (unless otherwise stated).

3.4

absorber

a device in which gaseous mercury (and mercury attached to small particles that are not filtered) is absorbed into an absorption solution

3.5

gas divider, impingers or fritted bubblers

a part in the absorber bottle which divides the gas stream in small bubbles into the absorption liquid

4 Principle

A sample stream of flue gas is extracted representatively from a duct or chimney over a certain period of time with a controlled flow and known volume. Dust in the sampled gas stream is collected on a filter whereafter the gas stream is passed through a series of absorbers, which contain an appropriate absorption solution for collecting gaseous mercury.

At the end of the sampling period the filter and absorption solution are collected to be taken to the laboratory.

The collected dust on the filter is digested in such a way that the mercury contained in the dust fraction is dissolved in a liquid. This liquid is then analyzed.

The absorption solution from the absorbers is prepared for analysis and analyzed.

The data from the sampling and analysis are combined and the results are expressed in milligrams of total mercury per cubic meter (mg/m^3) of flue gas.

The analysis of mercury is performed according to EN 1483.

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5 Sampling equipment

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5.1 Conditions for isokinetic and non-isokinetic sampling

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Although mercury is mainly present in the gaseous form, it can also be found in the dust phase as well as in droplets which can be present after wet scrubbers. Therefore isokinetic sampling is necessary in order to collect dust and droplets correctly.

When sampling has to be performed isokinetically then both dust and droplets, as well as flue gas, shall be sampled in one and the same equipment. This approach is necessary, due to the delicate gaseous/solid/droplet partitioning of mercury.

NOTE 1 As the total mercury content is to be determined, a shift in the equilibrium within a single sampling system does not influence the measurement of the total amount of mercury.

If the sum of the mercury content in the dust phase and droplets corresponds to less than $1 \mu\text{g}/\text{m}^3$, then gaseous mercury can be sampled non-isokinetically.

NOTE 2 An estimate of the amount of mercury in droplets may be determined on basis of the mercury content in the wet scrubber solution and the droplet content in the flue gas.

In the case that no droplets are present non-isokinetic sampling can also be performed when the ratio between the highest and lowest velocity at any point in the sampling plane is less or equal to a factor of 1,2.

5.2 General requirements

The sampling equipment consists of

- a probe with an entry nozzle and a temperature controlled tube;
- a filter housing with a filter support and a filter;

- a series of absorbers;
- suction unit(s) with gas metering device(s) and flow regulator(s).

The filter housing may be located:

- in the duct or chimney mounted directly behind the entry nozzle (in-stack filtration);
- outside the duct or chimney directly behind the suction tube (out-stack filtration).

In the latter case (out-stack filtration) the filter housing shall be temperature controlled.

NOTE The filter can also be placed semi-outstack, in the probe after a short piece of gas preheated suction tube.

5.3 Isokinetic sampling equipment

Reference is made to prEN 13284-1:1998 for the specific details about the requirements of the sampling equipment, including the equipment for the determination and maintaining of isokinetic conditions. In this present standard only specific requirements for the sampling of mercury (gaseous, dust and droplets) are stated, additional to those specified in prEN 13284-1:1998.

NOTE 1 The geometrical dimensions of glass nozzles will deviate from those specified in prEN 13284-1:1998, but the deviations can be decreased so that the remaining differences will not have a significant influence on the result.

Depending upon the type of absorbers used (see 5.5) two different sampling arrangements may be employed; a so-called 'main-stream arrangement' or a 'side stream arrangement'.

In the main-stream arrangement all the sampled flue gas is passed through the absorbers, but in the side-stream arrangement only a part of the sampled flue gas is passed through the absorbers.

Figures of both arrangements of the isokinetic sampling equipment for total mercury are given in Annex A.

The sampling probe shall be temperature controlled. If an out-stack filter is employed then this shall also be temperature controlled.

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NOTE 2 In cases of wet flue gases containing droplets or saturated flue gases it is advised to use out-stack filtration.

NOTE 3 In the case of the side-stream arrangement the connection to the T-piece and the T-piece itself shall be temperature controlled. Insulation of these parts, without additional heating, is not allowed.

NOTE 4 The heat input from the sampled gas is insufficient to compensate for the thermal losses even with insulation.

The parts of the equipment that are in contact with the sampled flue gas shall be made of specific materials which are prescribed in 5.9.

5.4 Non-isokinetic sampling equipment

In general the non-isokinetic sampling equipment is assembled according to the main-stream arrangement. Depending upon the gas flow employed, different types of absorbers (see 5.5) may be used.

A figure of the non-isokinetic sampling equipment is given in Annex A.

The sampling probe shall be temperature controlled. If an out-stack filter is employed then this shall also be temperature controlled.

The parts of the equipment that are in contact with the sampled flue gas shall be composed of specific materials which are prescribed in 5.9.

5.5 Absorbers

For efficient collection two absorbers shall be placed in series.

Downstream of these absorbers an extra empty absorber may be used as a liquid trap and as a protection for the downstream equipment.

The amount of mercury collected in the second absorber shall correspond to less than 5 % of the total amount of mercury in both absorbers or less than $2 \mu\text{g}/\text{m}^3$, whichever is the greatest.

Impingers or fritted bubblers can be used (figures of absorbers are contained in Annex B).

NOTE 1 Impingers have the advantage that they can be employed under high flow rate conditions. The disadvantage is the larger amount of (corrosive) absorption solution compared to that employed by fritted bubblers.

NOTE 2 Typical flow rates for impingers are in the range from $1 \text{ m}^3/\text{h}$ to $2 \text{ m}^3/\text{h}$ (17 l/min to 33 l/min).

NOTE 3 This standard is not validated for impingers.

NOTE 4 The advantage of fritted bubblers is a good absorption efficiency with a smaller amount of absorption solution than that employed by impingers. The disadvantage is that normally only a limited gas flow rate can be used (from about $0,06 \text{ m}^3/\text{h}$ (1 l/min) to approximately $0,18 \text{ m}^3/\text{h}$ (3 l/min)). If these bubblers are used in isokinetic sampling equipment a secondary sampling line (a side-stream arrangement) is needed.

5.6 Filter

5.6.1 Filter collection efficiency

For the filter collection efficiency the same specifications as in prEN 13284-1:1998 are required. This efficiency shall be certified by the supplier.

The filter material shall have an efficiency of 99,5 % on a test aerosol with a maximum abundance at a particle diameter of $0,3 \mu\text{m}$ at the maximum flow rate anticipated, (or 99,9 % on a test aerosol of $0,6 \mu\text{m}$ mean diameter), see prEN 13284-1:1998.

5.6.2 Filter material

The filter may be of any shape (flat, thimble or wool). The filter material shall be applicable up to the maximum temperature anticipated. The filter material shall have a blank value for mercury (calculated in units of $\mu\text{g}/\text{m}^3$ of sampled flue gas) of less than $0,1 \mu\text{g}/\text{m}^3$.

The filter material may consist of glass fibre, quartz fibre or polytetrafluoroethylene (PTFE), see 5.9.

When using filters with organic binders precautions shall be taken to ensure, that during digestion all mercury attached to the filter material will be dissolved. The organic binders or the reaction products from these binders after digestion shall not influence the analysis.

5.7 Connections

The choice of materials for the connections between the different parts of the sampling equipment are given in 5.9 and shall be employed to those parts which are in contact with the mercury containing flue gas.

For the main-stream arrangement these materials shall be employed from the nozzle to the last absorber.

For the side-stream arrangement these materials shall be employed from the nozzle to the last absorber in the side-stream.

The total length of unheated connections (such as tubing) from the sampling probe to the absorbers shall be as short as possible but less than 1 m.

The use of silicone tubing is limited in relation to the sampled gas flow rate: the total inner surface of all silicone tubing in the sampling equipment shall be less than $0,0033 \text{ m}^2$ per m^3/h (2 cm^2 per litre/min).

NOTE 1 Laboratory tests have shown severe losses in silicone tubing for Hg^{2+} if the surface to gas flow ratio is higher than 2 cm^2 per l/min.

NOTE 2 As an example of how this restriction may be applied: the total length of tubing with an inner diameter of 6 mm and a sampling gas flow of 1,5 l/min is limited to 1,6 cm.

5.8 Storage bottles and containers

The material of the storage bottles for the absorption solutions is prescribed in 5.9.

The storage bottles for the permanganate/sulphuric acid solution (2 % *m/m* KMnO_4 /10 % *m/m* H_2SO_4 , see 6.2) shall be darkened or stored in a dark place to avoid enhanced formation of MnO_2 .

The material of the filter storage containers is prescribed in 5.9.

5.9 Choice of materials for the sampling equipment

The parts of the sampling equipment in contact with the mercury containing flue gas, or with liquids containing mercury, shall be made of the materials shown in table 1. The different parts are described in 5.3 to 5.8.

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Table 1 — Materials for the sampling equipment

Equipment part	Material	Remark
Nozzle	Standard laboratory glass Borosilicate glass Quartz glass PTFE Titanium	
Sampling probe (inner suction tube)	Standard laboratory glass Borosilicate glass Quartz glass PTFE Titanium	
Filter housing Filter support	Standard laboratory glass Borosilicate glass Quartz glass PTFE Titanium	
Filter material	Flat filter: - quartz fibre - glass fibre - PTFE Thimble filter: - quartz fibre - glass fibre Wool filter: - quartz fibre - glass fibre	Efficiency see 5.6.1
Absorbers	Standard laboratory glass Borosilicate glass Quartz glass	
Connection fittings	Standard laboratory glass Borosilicate glass Quartz glass Titanium PTFE	Also the T-piece for the side-stream arrangement of the equipment (see also 5.7) Ball joints of the stated materials with PTFE lined seals are also allowed
Connection tubing	PTFE Silicone (with a total inner surface of less than 2 cm ² per l/min)	Use of silicone tubing is restricted (see 5.7)
Storage bottles	Standard laboratory glass Borosilicate glass Quartz glass Polypropylene (PP) Polyethylene (PE) ¹⁾	For solutions and samples
Cap of storage bottles	PTFE, PFA, FEP Polypropylene (PP) Uncoloured polyethylene (PE) ¹⁾	Caps of other materials are allowed as long as they have an insert of an allowed material
Filter storage container	Standard laboratory glass Borosilicate glass Quartz glass Polypropylene (PP) Polyethylene (PE) PTFE Titanium	

1) Only high density polyethylene (HDPE) shall be used.